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The Crystal and Molecular Structures of *syn*- and *anti*-Ethyl Benzohydroximate

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The crystal structures of *syn*- and *anti*-ethyl benzohydroximate, $C_6H_5-C(=NOH)-OC_2H_5$, have been determined, using three-dimensional diffractometer-collected X-ray data. Both compounds crystallize in the space group $P2_1/c$ with 4 molecules in unit cells with dimensions, respectively, of $a=12.001(9)$, $b=5.800(6)$, $c=17.44(1)$Å, $\beta=132.01(4)°$, and $a=11.52(1)$, $b=6.373(9)$, $c=15.87(1)$Å, $\beta=129.01(7)°$. The crystals of each compound are composed of dimers formed by pairs of OH···N hydrogen bonded molecules. The configurations about the C=N bonds agree with the assignment, based on dipole moment measurements.

The configurations of the two stereoisomeric forms of the ethyl esters of benzohydroximic acid, $C_6H_5-C(=NOH)-OC_2H_5$, were originally investigated by Werner,[1] but dipole moment and ^1H NMR studies, performed by Exner *et al.*,[2–4] indicated that the configurations proposed by Werner should be reversed. The present X-ray investigation was proposed by Exner as a means to obtain an unambiguous determination of the molecular structures. The research is also part of a conformational study of hydroxylamine derivatives, especially of hydroxamic acids, which have been found to possess numerous biological activities, *e.g.* inhibitory effect on DNA synthesis.[5] A preliminary description of the conformations of the molecules as determined by the X-ray diffraction method, and a comparison of these with the results deduced from the dipole moment measurements have been given recently.[6]

EXPERIMENTAL

Crystals suitable for X-ray study were kindly provided by O. Exner, the J. Heyrovský Institute of Polarography, Prague. The colourless, rod-shaped crystals of both compounds were volatile at room temperature, and it was necessary for the diffraction work to seal them in Lindemann glass tubes. Some crystal data for the compounds are listed in Table 1. The densities were measured by flotation in potassium iodide solution.

The lattice parameters were calculated from a series of diffractometer-measured θ-values. The intensity data were collected with a NONIUS 3-circle automatic dif-

Table 1. Crystal data for *syn*- and *anti*-ethyl benzohydroximate.

	syn	*anti*
Mol. formula	$C_9H_{11}NO_2$	$C_9H_{11}NO_2$
M.W.	163.20	163.20
M.p.	53 – 54°	67 – 68°
Space group	$P2_1/c$	$P2_1/c$
a	12.001 ± 0.009 Å	11.52 ± 0.01 Å
b	5.800 ± 0.006 Å	6.373 ± 0.009 Å
c	17.44 ± 0.01 Å	15.87 ± 0.01 Å
β	132.01 ± 0.04°	129.01 ± 0.07°
V	902.0 Å^3	905.4 Å^3
Z	4	4
D_x	1.201 g/cm^3	1.196 g/cm^3
D_m	1.207 g/cm^3	1.197 g/cm^3
μMo$K\alpha$	0.94 cm^{-1}	0.93 cm^{-1}
Cryst. size	0.2 × 0.3 × 1.0 mm^3	0.17 × 0.3 × 1.2 mm^3
Rot. axis	b	b

fractometer by the moving crystal-stationary detector technique, using crystal monochromatized Mo-radiation ($\lambda = 0.71069$ Å). Intensities of reflexions were measured in the range $3° < \theta < 25°$, and the intensity of a reference reflexion was measured for every 25 reflexions. Thus about 1500 independent reflexions were collected from each crystal. A reflexion was considered unobserved and was omitted when the intensity observed was less than 2.5 times its corresponding estimated standard deviation. Consequently, the numbers of observed reflexions were reduced to 802 and 834 for the *syn*- and *anti*-compounds, respectively. These data were corrected for Lorentz and polarization effects, but no corrections for absorption or extinction were made.

STRUCTURE DETERMINATION

Both structures were determined by a symbolic addition procedure for centrosymmetric crystals,[7,8] based on the satisfaction of the $\sum_2$ relationship.[9]

syn-*Ethyl benzohydroximate.* Four strong reflections, 1 $\bar{2}$ 4, 2 $\bar{3}$ 10, 0 $\bar{2}$ 5, and $\bar{2}$ $\bar{5}$ 5, all with $|E| > 3$, were assigned symbols to represent their phases, and were used as "knowns" to implement phase determination, using the $\sum_2$ relationship between the 330 observations with $|E| > 1.16$. After six cycles of the usual iterative procedure, 163 signs had been determined, either directly or in terms of the "known" starting symbols. The reflexions 1 $\bar{2}$ 4, 2 $\bar{3}$ 10, and $\bar{2}$ $\bar{5}$ 5 were given plus-sign to specify an origin, while the phase of the 4th reflexion, 0 $\bar{2}$ 5, was assigned to be minus, this combination of the four signs being much more probable than the "all plus" combination.

The E-map computed, using the 163 terms, whose phases were determined from this assignment, revealed the positions of all 12 non-hydrogen atoms of the structure. The coordinates of these atoms, as read from the E-map, were subjected to a Fourier refinement, during which the conventional R-value dropped from 0.56 to 0.32. The atomic scattering factors were taken from *International Tables*.[10] This refinement was followed by 3+3 cycles of full matrix least squares refinement, in which positional parameters as well as individual atomic thermal parameters were varied, isotropic and anisotropic, respectively. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where

Table 2. Observed and calculated structure factors for *syn*-ethyl benzohydroximate. The columns are l, $10 \times |F_o|$, and $10 \times F_c$.

H,-5,0

-2	75	-86
-1	153	150

H,-5,1

2	39	47
0	36	-27
-3	33	-17

H,-5,2

-4	31	17
-2	35	-30
-1	132	146
0	57	-65
1	102	107

H,-5,3

0	88	-98
-1	60	-63
-2	120	123
-3	42	36
-4	40	-42

H,-5,4

-5	84	78
-4	73	-73
-3	32	42
-2	74	-82
-1	52	-54

H,-5,5

0	34	37
-2	160	169
-3	67	71
-4	80	-73
-5	66	63

H,-5,6

-3	87	99
-2	32	-24
-1	57	-57

H,-5,7

-4	102	-112

H,-4,11

-7	44	38
-6	53	54
-5	66	-68

H,-4,10

-2	52	60
-3	151	-145
-4	43	38
-5	47	51
-7	50	-51

H,-4,9

-7	47	-48
-6	53	64
-4	67	-77
-3	144	157
-2	47	56

H,-4,8

0	32	-24
-1	45	50
-3	196	-200
-5	38	-21
-6	58	-69
-8	103	-110

H,-4,7

-8	54	-58
-4	48	-43
-3	50	46
-2	62	-58
-1	70	-72
0	72	-73

H,-4,6

-1	181	182
-2	71	73
-5	56	-55
-6	74	77

H,-4,5

-7	44	46
-6	95	105
-5	87	-99
-4	45	50
-3	39	-32
1	32	32
2	57	-58

H,-4,4

3	81	88
2	97	-105
0	71	-76
-1	46	45
-2	55	46
-3	136	134
-4	36	21
-5	71	-72
-6	156	162

H,-4,3

-6	103	108
-5	70	-68
-4	85	76
-3	110	102
-1	32	-27
0	125	115
1	164	-164
2	34	33
3	53	53

H,-4,2

3	54	58
2	100	-109
1	107	107
0	124	-115
-1	143	-147
-2	61	56
-5	80	-80
-6	106	107

H,-4,1

-5	58	61
-4	58	-62
-3	69	73
-1	290	-283
0	166	164
2	32	-32
3	132	137
4	72	78
5	108	-114

H,-4,0

0	117	-118
-1	54	48
-2	54	58
-3	40	-48

H,-3,0

-5	124	-120
-4	129	125
-3	184	-177
-2	261	-226
-1	68	-47

H,-3,1

4	70	-72
3	123	131
2	69	-71
1	285	-254
0	163	138
-1	36	-22
-2	214	-186
-3	320	303
-4	60	-59
-5	44	-54
-7	81	80

H,-3,2

-7	129	138
-4	151	141
-3	41	38
-1	86	-92
0	280	-261
1	176	169
2	33	-44
3	105	100
5	76	80
6	45	-31

H,-3,3

-4	59	66
-5	76	83
-6	114	-111
-8	34	58

H,-3,4

-7	212	231
-6	39	-43
-5	58	48
-4	103	100
-3	193	170
-2	39	35
-1	51	-45
0	85	95
2	56	-51
3	53	61
5	37	45

H,-3,5

3	104	-102
2	133	135
0	32	-38
-1	81	-86
-2	157	145
-3	159	-148
-5	69	63
-8	65	-67
-9	37	-26

H,-3,6

-9	78	-83
-8	112	-114
-6	55	-44
-3	106	-99
-1	137	-142
0	70	-68
1	72	-70
2	86	89
3	82	-81

H,-3,7

0	57	-52
-2	108	109
-4	69	-69
-5	50	40
-6	45	49
-7	109	-108
-9	25	-4

H,-3,8

-9	57	-63
-8	88	-95
-5	36	-41
-3	42	-34
-2	183	176
-1	163	-165
0	91	-100

H,-3,9

1	38	-49
-1	139	137
-2	48	-59
-3	71	65
-4	98	-101
-6	59	64
-7	35	-35

H,-3,10

-9	33	-40
-8	50	52
-5	135	136
-4	37	-37
-3	60	57
-2	217	229

H,-3,11

-1	97	106
-2	127	-123
-5	32	32
-6	47	-62
-7	63	71
-8	50	-50
-9	30	-32

H,-3,12

-8	61	-64
-7	60	-56
-5	57	51
-4	148	-144
-3	35	18
-2	41	34

H,-3,13

-4	59	66
-5	76	83
-6	114	-111
-8	34	58

H,-3,14

-6	62	-63

H,-2,15

-9	76	72
-8	44	-37
-7	84	-93
-6	94	95
-5	58	-58

H,-2,14

-3	109	107
-4	39	40
-5	61	-60
-6	44	42
-8	39	-34
-9	33	36

H,-2,13

-10	34	-38
-8	95	93
-7	149	-154
-4	52	-57
-3	63	-59
-2	114	111

H,-2,12

-1	72	-62
-2	69	-64
-6	61	63
-7	170	-162
-8	38	47
-9	49	-35
-10	47	-43

H,-2,11

-10	57	62
-9	44	-32
-7	30	-12
-6	86	87
-3	69	-67
-1	61	65
0	66	-71

H,-2,10

0	59	58
-2	95	95
-3	111	-116
-4	46	36
-5	31	27
-6	38	27
-7	30	-35
-8	38	48
-9	41	-49
-10	72	-74

H,-2,9

-9	44	-36
-8	90	-96
-6	154	151
-5	42	-45
-3	62	59
-1	159	157
0	112	-113

H,-2,8

2	49	52
-1	131	133
-2	190	181
-3	87	79
-5	158	148
-6	78	66
-7	68	64
-9	113	119
-10	50	-51

H,-2,7

-9	120	-126
-6	71	71
-5	150	-160
-4	131	-123
-3	256	255
-2	80	-78
0	159	-158
2	74	-67

H,-2,6

4	37	-19
1	38	27
0	186	-189
-1	154	-152
-2	205	-207
-3	95	97
-4	262	-235
-5	154	145
-6	71	-86

H,-2,5

-9	104	-99
-8	50	45
-7	50	-47
-6	115	106
-5	95	-89
-4	51	-46
-3	230	211
-1	305	-304
0	354	-364
1	126	136
2	83	-81
3	82	-71
4	53	48
5	34	-33

H,-2,4

2	153	-148
1	416	419
0	29	-20
-1	217	-204
-2	78	-70
-3	31	-8
-4	259	-264
-6	31	40
-7	48	-37
-9	65	-64

H,-2,3

-9	72	-75
-8	118	119
-7	31	36
-6	30	39
-5	68	76
-4	175	163
-2	126	-109
-1	130	116
0	205	-193
1	81	82
2	332	322
3	154	-150
4	104	101
6	35	45

H,-2,2

6	107	-107
5	49	46
4	67	64
3	115	-117
2	158	-143
1	201	190
0	82	72
-1	93	61
-2	417	371
-3	109	-111
-4	113	112
-5	51	-52
-6	53	64
-7	35	37
-8	109	109

H,-2,1

-8	67	69
-7	125	124
-4	56	62
-3	146	128
-2	373	-324
-1	103	103
0	266	211
1	53	-52
2	410	396
3	108	-106
4	136	130
5	48	50
6	77	81
7	35	-33

H,-2,0

0	202	161
-1	335	-276
-2	73	70
-3	111	-112
-4	148	154
-5	60	-65
-6	96	-100
-8	41	37

H,-1,0

-8	92	86
-4	55	-45
-3	147	-152
-2	364	382
-1	163	-175

H,-1,1

6	51	-49
5	52	-53
4	263	264
3	77	-77
2	387	-395
1	1142	1226
0	40	-51
-1	174	244
-2	27	-33
-3	116	107
-4	59	-57
-5	120	117
-6	125	-115
-7	29	30
-8	56	55

H,-1,2

-6	148	141
-5	81	90
-4	83	-89
-3	119	115
-2	222	208
-1	295	-296
0	530	511
1	618	638
2	115	-111
3	242	249
5	65	69
6	87	90
7	42	36

H,-1,3

5	80	-88
4	78	81
3	66	-67
2	164	-156
1	149	149
0	346	355
-1	589	-574
-2	382	-363
-3	157	160
-4	158	-156
-5	114	-118
-6	74	-71

H,-1,4

-8	88	-83
-7	51	55
-6	40	-35
-5	137	-138
-4	55	48
-3	169	174
-2	407	-411
-1	618	-642
1	140	-155
3	137	143
4	109	-104
6	45	38

H,-1,5

5	50	36
3	77	-70
2	72	65
0	28	-24
-1	413	-419
-2	34	55
-3	278	-292
-5	368	-365
-6	194	196
-7	48	-58
-9	60	61
-10	59	-58

H,-1,6

-10	47	-37
-7	121	119
-5	41	-44
-4	35	39
-3	119	129
-2	209	208
-1	136	-138
0	145	-143
1	200	-202
2	37	27
3	30	-21
4	50	-45

H,-1,7

3	47	45
1	128	131
0	56	-57
-1	75	-81
-2	129	143
-3	250	-249
-4	343	335
-5	213	-210
-6	184	192
-7	90	79
-8	36	40
-10	34	18

H,-1,8

-11	31	-26
-10	32	-28
-9	48	-59
-8	83	-89
-7	73	73
-6	87	74
-4	127	-127
-2	239	248
-1	50	-57
0	93	97

H,-1,9

1	64	69
0	106	-114
-2	62	54
-3	36	-42
-4	93	101
-5	285	276
-6	37	-46
-8	147	152
-9	31	-8
-10	44	-61

H,-1,10

-8	112	-111
-7	59	-61
-6	58	54
-5	39	38
-4	135	-130
-3	45	47
-1	70	-73
0	56	53
1	44	-36

H,-1,11

-1	39	-38
-3	100	99
-4	41	-27
-5	49	42
-6	38	-27
-8	78	-75
-9	88	84
-10	153	-147

H,-1,12

-11	45	-39
-10	96	105
-9	114	112
-8	111	-110
-7	48	53
-6	55	62
-5	29	-35
-3	122	117
-2	38	-33
-1	34	39

H,-1,13

-1	45	-44
-5	74	-78
-6	83	-75
-7	100	99
-8	120	-110
-10	114	-99

H,-1,14

-10	77	74
-9	38	-48
-8	67	-68
-7	115	120
-6	82	-91
-5	40	-47

H,-1,15

-5	65	-66
-8	57	-53
-9	44	-40

H,-1,16

-9	82	-87
-6	66	-61

H,0,16

-5	43	40
-9	34	-25

H,0,14

-4	66	-59
-5	41	-24
-6	57	-63
-7	30	-16
-8	34	38
-9	227	-229
-10	29	-20
-11	69	69

H,0,12

0	77	-76
-1	81	75
-2	33	23
-4	124	116
-5	41	-44
-6	119	-127
-8	196	194
-9	272	-272
-11	34	-30

H,0,10

2	76	73
1	103	-96
0	63	-62
-2	92	-91
-3	57	59
-4	392	396
-5	121	-115
-7	157	162
-8	37	47
-9	77	-77
-10	105	98
-11	51	-39

H,0,8

3	68	-65
2	169	172

Table 2. Continued.

H,0,8
0 47 58
-1 102 -110
-2 212 -212
-3 202 204
-4 214 -220
-5 143 152
-6 193 188
-8 85 -80
-9 107 94
-10 46 -59
-11 47 -42

H,0,6
5 64 66
4 38 34
3 51 -71
2 94 106
1 138 144
-1 122 140
-2 138 -154
-3 188 159
-4 465 -475
-5 430 454
-6 187 -192
-7 280 296
-8 104 91
-9 42 -32

H,0,4
6 129 -121
5 67 76
3 129 -129
2 64 -64
1 131 136
0 439 -469
-1 358 -420
-2 335 -350
-3 67 58
-4 194 -193
-5 220 -211
-6 235 -242
-7 155 158
-8 29 30
-10 88 75

H,0,2
6 120 -123
5 180 175
4 187 -159
3 38 -32
2 470 472
1 253 -254
-3 148 -158
-4 315 336
-5 351 -361
-6 94 -88
-8 30 -7

H,0,0
-2 55 -61
-3 402 438

-4 84 66
-5 32 -35
-6 84 77

H,-6,0
-1 43 -55
0 66 -71
1 40 -55

H,-6,1
1 67 -69
0 83 85
-1 73 64
-3 61 -66

H,-6,2
-6 43 32
-4 38 33
-3 34 52
4 39 -28

H,-6,3
1 35 -37
0 67 76
-2 46 -32
-3 83 -96
-4 91 97

H,-6,4
-4 42 35
-2 37 32
-1 75 85

H,-6,5
2 35 -28
-3 45 -44
-4 89 86
-6 49 -49

H,-6,6
-2 45 47
0 35 -42

H,-6,7
-5 37 46
-6 50 -50

H,-6,9
-4 52 -55
-5 70 58

H,-5,13
-9 36 48
-6 45 41

H,-5,12
-7 37 -39

H,-5,11
-9 33 32
-8 52 63

H,-5,10
-3 37 -49
-7 39 -54
-9 41 -28

H,-5,9
-7 90 -94
-4 56 -60
-2 38 -47

H,-5,8
0 35 30
-4 59 57
-5 82 -98
-6 63 69
-8 39 -46

H,-5,7
-7 80 -90
-6 39 36
-5 42 46
-1 60 54
0 33 25

H,-5,6
2 35 -28
0 37 20
-6 70 78

H,-5,5
1 36 -34

H,-5,4
2 74 -72
-6 38 -43

H,-5,3
-6 36 -43
-5 64 92
3 34 -43
5 33 -27

H,-5,2
-9 77 66
-6 53 -36
4 65 62

H,-5,0
-4 41 52

H,-4,0
-6 34 36

H,-4,1
6 56 60
-8 39 37

H,-4,2
5 34 -31
7 38 32

H,-4,4
-8 37 -17
4 46 -56

H,-4,5
-9 41 -51

H,-4,12
-9 58 66

H,-4,13
-3 82 -77
-4 33 30

H,-4,14
-11 47 -27
-6 32 -38
-3 34 -37

H,-4,15
-6 33 -36

H,-4,16
-7 35 32
-6 45 -38
-5 36 30

H,-4,6
-8 102 -102
3 37 35

H,-4,7
4 39 -37
2 44 -40
-9 71 -70

H,-4,9
-8 56 -61
-9 66 81

H,-4,10
-11 39 36
-9 71 77

H,-4,11
1 39 -36
-1 60 63
-8 83 -79
-9 47 46

H,-3,1
7 46 -34

H,-3,6
5 46 32

H,-3,8
3 56 -51

H,-3,10
-10 59 51

H,-3,12
-1 47 42

H,-3,13
-3 59 -55
-9 40 -46

H,-3,14
-10 39 35
-8 48 -39
-4 43 -42
-3 33 -34
-2 42 -29

H,-3,15
-7 60 -55
-8 78 88
-11 34 32
-12 57 -46

H,-3,16
-8 51 49
-7 41 -34
-5 36 40

H,-3,17
-6 54 42
-7 39 -44

H,-2,2
8 46 -29

H,-2,7
4 43 -22

H,-2,9
-11 51 52

H,-2,11
1 40 30
-11 86 93
-12 44 -34

H,-2,12
-12 55 53

H,-2,13
-11 73 72

H,-2,14
-13 33 -34
-12 37 32
-11 35 -15
-2 45 -44

H,-2,15
-4 64 -63

H,-2,16
-8 63 -62
-7 69 69
-5 35 -42

-3 44 29

H,-2,17
-7 34 26
-8 62 -62
-9 58 60

H,-2,18
-8 48 -43

H,-2,19
-7 41 23
-9 34 32

H,-1,0
-10 38 -27

H,-1,1
8 54 57

H,-1,2
-10 53 -46

H,-1,3
7 94 -85
-11 39 27

H,-1,5
7 45 -45

H,-1,7
5 56 51
-11 35 -42

H,-1,9
3 49 44

H,-1,10
-12 54 59
4 39 -31

H,-1,11
-12 43 38

H,-1,12
-12 41 42
1 56 -57

H,-1,13
1 38 -29
0 37 25

H,-1,14
-11 58 -63
-1 83 79

H,-1,16
-10 37 45
-5 43 38
-4 41 -28
-3 70 -68

H,-1,17
-2 35 19
-7 35 41
-9 40 -32
-10 94 83

H,-1,18
-10 54 48
-5 43 44

H,-1,19
-10 38 38

H,0,2
-10 45 40
9 53 48

H,0,4
7 49 -59
-11 77 -75

H,0,6
-11 40 -52
7 44 -32

H,0,8
6 46 38
-12 45 37

H,0,10
-13 51 44
3 46 -29

H,0,14
0 42 -44
0 44 -44
1 52 34

H,0,16
-13 33 -14
-11 160 148
-3 49 46

H,0,18
-13 33 9
-12 56 -41
-11 49 46
-9 35 39
-6 61 56
-4 35 -34

H,0,20
-12 47 -29

H,0,0
-9 65 52

$w = 1/(A + B|F_o| + C|F_o|^2)$. The coefficients A, B, and C were chosen in such a way that reflexions with $26 < |F_o| < 100$ were given unity weight, and reflexions with $|F_o| < 26$ and $|F_o| > 100$ were given weights less than 1.0.

The R-value was reduced to 0.102, and at this stage, a difference Fourier map was computed. It revealed unambiguously the positions of all 11 hydrogen atoms. Although the contributions of the hydrogen atoms to the scattered amplitudes were now included, the parameters of these atoms were not varied during 2 further cycles of least squares refinement of the heavier atoms. The hydrogen atoms were given isotropic temperature factors of 4.0 Å^2. The final R-value was 0.072.

Observed and calculated structure factors are listed in Table 2. Table 3 lists the fractional coordinates of the heavier atoms and the approximate fractional coordinates of the hydrogen atoms. Table 4 lists the final thermal parameters of the non-hydrogen atoms.

anti-*Ethyl benzohydroximate*. Five strong reflexions, $\bar{9}$ 0 12, $\bar{2}$ 3 10, 2 2 1, $\bar{10}$ 1 11, and $\bar{7}$ 2 13, with $|E| > 2.3$, were assigned symbols to represent their

Table 3. Fractional atomic coordinates for *syn*-ethyl benzohydroxymate.

	x	*y*	*z*
C(1)	0.7695 (6)	0.6100 (9)	0.5306 (4)
C(2)	0.9029 (7)	0.5886 (10)	0.5531 (5)
C(3)	1.0044 (7)	0.4134 (13)	0.6212 (5)
C(4)	0.9670 (9)	0.2674 (11)	0.6642 (5)
C(5)	0.8345 (8)	0.2922 (10)	0.6410 (5)
C(6)	0.7324 (7)	0.4632 (10)	0.5741 (4)
C(7)	0.6627 (7)	0.7971 (9)	0.4580 (4)
N(8)	0.5777 (5)	0.8785 (8)	0.4712 (3)
O(9)	0.4684 (4)	1.0374 (7)	0.3941 (3)
O(10)	0.6676 (5)	0.8428 (6)	0.3858 (3)
C(11)	0.6387 (8)	1.0754 (10)	0.3423 (5)
C(12)	0.7036 (8)	1.0791 (12)	0.2928 (6)
H(2)	0.930	0.703	0.517
H(3)	1.112	0.395	0.642
H(4)	1.044	0.130	0.716
H(5)	0.806	0.175	0.674
H(6)	0.625	0.482	0.555
H(9)	0.395	1.106	0.405
H(111)	0.518	1.109	0.285
H(112)	0.689	1.206	0.401
H(121)	0.681	1.252	0.261
H(122)	0.635	0.940	0.243
H(123)	0.820	1.090	0.360

Table 4. Thermal parameters for *syn*-ethyl benzohydroxymate.
$T_i = \exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	4.7 (3)	2.6 (3)	4.3 (3)	0.1 (3)	3.0 (2)	−0.5 (2)
C(2)	5.3 (4)	3.8 (3)	6.7 (3)	0.7 (3)	4.2 (3)	0.5 (3)
C(3)	6.0 (4)	5.7 (4)	7.4 (4)	1.6 (4)	4.4 (4)	0.9 (4)
C(4)	8.1 (5)	3.5 (3)	6.8 (4)	1.1 (3)	5.2 (4)	1.2 (3)
C(5)	7.1 (4)	3.2 (3)	5.8 (3)	−0.6 (3)	4.5 (4)	−0.2 (3)
C(6)	6.1 (4)	2.9 (3)	5.1 (3)	−0.2 (3)	3.9 (3)	−0.1 (3)
C(7)	5.3 (4)	3.2 (3)	4.5 (3)	−0.1 (3)	3.8 (3)	−0.7 (2)
N(8)	5.5 (3)	3.0 (2)	5.4 (3)	0.0 (2)	3.8 (2)	−0.2 (2)
O(9)	6.1 (2)	4.2 (2)	6.6 (2)	1.8 (2)	4.4 (2)	0.8 (2)
O(10)	8.1 (3)	3.0 (2)	6.0 (2)	1.2 (2)	5.5 (2)	0.6 (2)
C(11)	9.5 (4)	3.1 (3)	8.6 (4)	1.4 (3)	7.0 (4)	1.9 (3)
C(12)	9.6 (5)	4.6 (4)	11.2 (5)	0.6 (4)	8.2 (5)	2.0 (4)

phases. These were used as "knowns" in a phase determining process, analogous to that described in the preceding section. 420 reflexions with $|E| > 1.0$ were used for investigating $\sum_2$ relationships, and, after eight iterations, signs or symbol combinations representing signs had been allotted to 232 of these. The reflexions $\bar{9}$ 0 12, $\bar{10}$ 1 11, and $\bar{7}$ 2 13 were given plus-sign to specify the origin, following which signs of minus and plus for $\bar{2}$ 3 10 and 2 2 1, respectively, were indicated as being the most probable.

Table 5. Observed and calculated structure factors for *anti*-ethyl benzohydroximate. The columns are l, $10\times|F_o|$, and $10\times F_c$

H,0,0

2	237	-270
3	637	-706
4	54	-71
5	138	-144
9	30	28
10	31	34

H,0,2

-8	28	-32
-7	82	-62
-6	309	-315
-5	72	76
-4	244	255
-3	78	75
1	126	141
2	155	176
3	122	-137
4	206	-211
5	105	-94
6	28	-34
7	46	48

H,0,4

-10	59	71
-9	55	-38
-7	112	-118
-6	289	-323
-5	161	178
-4	36	-16
-3	223	240
-2	31	-50
-1	624	810
0	255	-257
2	71	-76
4	83	-85
5	28	-36

H,0,6

-8	62	54
-7	80	-55
-5	62	-76
-4	85	-96
-3	74	84
-2	166	164
-1	31	-51
0	58	-67
1	94	-112
2	106	119
5	88	-98

H,0,8

-10	31	-59
-9	48	-47
-7	103	121
-6	295	317
-4	140	-165
-3	251	-263
-2	163	-162
-1	32	28
2	77	99
3	30	37

H,0,10

-10	62	65
-9	52	61
-7	74	-85
-6	44	60
-5	69	77
-4	130	156
-3	206	-231
-2	156	-160
0	24	-45
1	113	117

H,0,12

-9	180	230
-8	111	146
-6	124	-144
-5	25	-17
-4	31	-27
-3	94	-102
-1	42	-61

H,0,14

-9	133	174
-6	65	-75
-4	48	-47

H,1,13

-10	43	45
-9	28	-27
-8	88	86
-7	40	47
-6	28	35
-5	96	-84
-4	41	-33
-2	36	-35

H,1,12

-1	70	61
-2	29	24
-3	50	-45
-4	26	-25
-5	28	-31
-7	66	-69
-8	106	-100
-9	51	-52
-10	132	126

H,1,11

-10	107	105
-9	55	53
-8	41	35
-7	34	-30
-6	35	-32
-5	49	-47
-4	39	-41
-2	38	-37
0	40	52

H,1,10

0	75	78
-1	120	114
-2	64	66
-4	219	-218
-5	120	-117
-7	35	27
-8	106	-98
-9	35	43
-10	74	69

H,1,9

-10	36	37
-8	98	-90
-7	72	80
-6	77	73
-5	226	209
-4	141	-133
-3	110	-98
-2	171	-154
0	87	86

H,1,8

0	97	96
-1	34	-29
-2	218	206
-3	161	-149
-4	290	-280
-5	95	-77
-6	114	114
-9	50	46
-10	25	-28

H,1,7

-7	169	139
-6	38	-42
-5	91	-79
-4	223	-216
-3	52	-51
-2	69	-85
0	80	79
1	55	60
2	35	-34
3	28	24
4	27	16

H,1,6

5	33	35
4	24	-21
1	157	165
0	154	-180
-1	18	14
-2	98	101
-3	204	-205
-5	43	30
-7	28	-20
-8	72	70
-10	29	-34

H,1,5

-10	27	17
-9	41	42
-6	41	-37
-5	243	-238
-4	165	157
-3	19	16
-2	240	225
-1	275	-284
0	207	222
1	132	149
2	57	-58
3	69	-72
4	26	-34
5	52	55

H,1,4

6	34	28
5	102	111
4	35	-19
3	64	-74
2	149	-151
1	25	19
0	159	-198
-1	401	437
-2	493	-459
-3	60	-62
-4	258	245
-5	256	302
-7	60	-58
-8	65	-55
-9	25	-27

H,1,3

-9	37	43
-7	56	-49
-6	25	-27
-5	233	-219
-4	71	57
-3	189	166
-2	486	479
-1	440	-430
0	122	-141
1	176	161
2	94	81
3	122	-123
4	31	-27

H,1,2

7	74	-79
5	49	51
4	163	158
3	195	-202
2	338	-355
1	772	-825
0	409	395
-1	276	260
-2	255	239
-3	62	72
-4	158	166
-5	76	72
-6	116	106
-7	100	-105
-8	61	-46
-9	61	-54

H,1,1

-7	36	-26
-6	32	28
-4	171	-157
-3	100	-90
-2	263	-236
-1	128	120
0	64	-67
1	946	1085
2	250	254
3	181	-182
4	297	-289
5	39	-45
6	33	33
7	45	-55

H,1,0

-1	426	418
-2	541	539
-3	203	178
-4	114	-105
-5	31	32
-7	27	-26

H,2,0

-8	58	49
-7	85	82
-6	89	-94
-4	61	61
-3	63	68
-2	22	-27
-1	38	16
0	151	-151

H,2,1

7	71	69
6	90	-101
5	101	97
3	37	33
2	507	-502
1	272	-263
0	229	-184
-1	212	203
-2	488	461
-3	179	163
-4	42	44
-6	29	31
-7	40	-34
-8	139	-140

H,2,2

-8	97	102
-5	20	11
-3	121	-116
-2	89	99
-1	456	-405
0	213	-206
1	236	-223
2	42	41
3	354	351
4	173	178
5	73	-70
6	122	-119
7	29	28

H,2,3

5	78	73
3	113	-102
2	308	-300
1	151	-152
0	112	104
-1	60	61
-2	144	123
-3	189	174
-4	23	21
-5	157	140
-6	26	-30
-8	174	-165

H,2,4

-7	68	64
-6	75	67
-4	263	-251
-2	115	-107
-1	297	290
0	91	87
1	291	-284
3	114	104
4	66	72
5	35	-38

H,2,5

5	36	-26
3	109	-111
2	61	53
0	216	210
-1	365	-371
-2	42	-42
-3	303	278
-4	160	150
-5	55	-49
-6	99	-94
-8	27	-26
-9	43	-41

H,2,6

-9	48	41
-8	46	34
-7	63	61
-6	108	-93
-5	134	-115
-4	198	-188
-2	96	-104
-1	316	304
0	61	-55
1	40	50
2	83	77
4	53	-49

H,2,7

2	32	35
0	93	82
-2	52	-46
-3	144	141
-5	244	-229
-6	70	-60
-7	99	101
-8	82	84
-9	102	-100

H,2,8

-9	56	-48
-8	111	110
-6	31	20
-5	182	-170
-3	125	-115
-2	212	205
0	47	-57
1	35	36
2	41	39

H,2,9

0	34	37
-1	171	168
-2	66	-62
-3	38	40
-4	32	-20
-5	103	-93
-6	152	-141
-8	60	59
-9	46	-41
-10	36	37

H,2,10

-10	61	-57
-8	33	-20
-7	59	-55
-6	42	-41
-5	67	-64
-3	132	127
-2	135	140
-1	34	-26
0	34	-36

H,2,11

0	45	41
-1	163	148
-2	33	27
-3	61	-54
-4	61	-53
-5	40	-47
-6	79	-75
-7	62	-60

H,2,12

-10	70	-69
-8	46	-29
-7	85	76
-5	43	-38
-4	67	-63
-3	49	44
-2	28	20
-1	73	66

H,2,13

-3	73	-74
-4	46	31
-5	42	33
-7	148	-128
-8	64	-58
-9	34	30

H,2,14

-8	42	-35
-7	41	30
-6	79	75
-5	57	53

H,3,13

-6	94	81
-5	37	45

H,3,12

-3	92	91
-4	114	-104
-5	46	-44
-7	77	73

H,3,11

-9	62	-54
-8	35	-35
-6	53	-47
-4	37	21
-3	76	73
-2	140	128

H,3,10

0	40	-39
-1	126	-121
-2	227	220
-3	49	48
-4	32	31
-5	90	-83
-7	55	-49

H,3,9

-8	31	-23
-7	47	-37
-6	96	-95
-4	105	109
-3	131	137
-2	32	32
0	46	-49
1	43	-37

H,3,8

2	32	30
0	97	-93
-1	43	49
-2	137	135
-4	24	20
-5	35	36
-6	28	-16
-7	60	-58
-8	145	-135
-9	39	42

H,3,7

-9	38	34
-8	69	65
-7	50	-59
-6	66	-63
-5	26	-25
-4	28	31
-2	55	-50
0	89	89

H,3,6

0	58	-58
-1	151	135
-2	93	91
-3	124	126
-4	50	-47
-6	98	-95
-7	93	-88
-8	64	-65
-9	118	116

H,3,5

-8	27	31
-5	67	59
-4	23	9
-3	149	-141
-2	76	-69
-1	71	59
0	131	132
1	45	43
2	67	-67
3	40	-41

H,3,4

4	51	-50
3	51	-51
2	96	-91
1	97	94
0	105	101
-1	95	90
-2	170	147
-3	120	-118
-4	142	130
-5	60	-57
-6	68	-62
-7	236	-242
-8	105	100

H,3,3

-8	43	-35
-7	128	130
-6	93	86
-5	95	92
-3	66	-63
-2	90	-80
-1	45	-34
0	221	-207
1	192	-190
2	25	-25
3	120	120
4	120	125
5	53	57

H,3,2

3	73	-60
2	76	66
1	62	52
0	197	-162
-1	149	135
-4	176	162
-5	26	-34
-6	146	-147
-7	111	-115
-8	111	108

H,3,1

-7	105	89
-6	124	123
-3	80	80
-1	263	-265
0	394	-354
1	246	-224
2	216	191
3	218	224
4	94	95
5	46	-46

H,3,0

-1	40	-32
-2	386	-377
-3	179	169
-4	121	107
-5	88	89
-6	123	-119

H,4,0

-6	73	70
-5	33	23
-4	50	-49
6	51	-49
5	78	-80
4	68	-69
3	177	177
2	167	162
1	170	169
0	92	-83
-1	240	-259
-5	32	-38

H,4,2

-6	86	84
-5	127	135
-4	41	43
-2	96	-94
0	171	-161
1	43	-57
5	51	63

H,4,3

4	48	-56
3	46	48
2	54	61
1	26	-23
-1	38	48
-2	123	115
-3	113	102
-4	187	-190
-5	141	-150
-6	46	-49
-7	94	96

H,4,4

-7	100	-87
-6	74	71
-5	111	113
-4	142	141
-3	45	-44
-2	79	75
-1	35	-36
0	74	-67
1	24	13
2	104	-106
3	67	-70

H,4,5

3	49	-53
2	26	32
0	62	63
-1	29	28
-2	98	95
-3	100	93
-4	80	-82
-5	127	-116
-6	87	-96
-7	86	79

H,4,6

-8	55	-55
-7	66	-59
-6	56	56
-5	89	94
-4	59	59
-2	52	55
-1	132	-127
0	78	88
2	50	-58

H,4,7

1	28	-19
0	62	57
-3	42	45
-4	61	69
-7	42	-42

H,4,8

-8	93	-92
-7	35	31
-5	104	105
-4	58	54
-3	120	120
-2	124	-129
-1	71	-74

H,4,9

-1	33	-26
-2	133	-131
-3	114	114
-4	163	165
-5	73	81
-6	71	-73
-7	110	-103

H,4,10

-6	30	-26
-4	110	106
-3	104	109
-2	32	-18

Table 5. Continued.

H,4,11			H,6,0			-11	46	51	H,2,9			-2	37	39	0	56	-47	H,5,6		
-4	46	55	0	54	-77				-11	53	45	-11	34	-24				3	34	33
-6	49	-43	1	79	105	H,1,12									H,4,11					
						0	51	48	H,2,10			H,3,11			0	40	-29	H,5,4		
H,5,8			H,6,1			-11	58	50	-12	55	45	-12	39	28	-2	42	-43	-7	48	44
-3	89	-85	1	53	-57	-12	37	42	-11	54	38	-11	40	17	-8	71	68			
-5	98	109	0	71	-83	-13	32	-16	2	30	-23	0	53	-48	-9	40	37	H,5,3		
			-1	42	45							1	68	-60	-10	83	-70	-7	69	-73
H,5,7						H,1,11			H,2,11						-11	50	-36	6	48	52
-6	44	49	H,6,2			-12	51	-61	2	43	-32	H,3,10								
-5	61	68	-2	47	55				-11	85	79	2	45	35	H,4,12			H,5,2		
-4	66	74	-1	63	-77	H,1,10						-11	31	-28	-10	34	24	4	67	64
-3	53	-49	0	37	-25	-11	42	41	H,2,12						-7	27	-22	-7	38	42
-1	30	25				-12	30	33	-12	43	42	H,3,9			-4	41	30	-8	39	40
			H,C,C						1	31	-20	-10	28	-33						
H,5,6			9	30	28	H,1,9			2	37	-30				H,4,13			H,6,0		
-1	80	83	10	40	34	4	33	43				H,3,8			-3	73	-64	-4	48	-60
-3	155	-154							H,2,13			4	49	-43	-6	38	34			
-4	48	-53	H,C,2			H,1,8			0	29	24				-7	28	15	H,6,1		
-5	52	51	-10	64	73	-11	32	39	-1	61	57	H,3,7						5	31	34
-6	81	84							-10	46	37	-11	41	-23	H,4,14			3	47	57
			H,C,4			H,1,7			-11	76	74				-8	36	-24			
H,5,5			7	73	71	-11	36	-49				H,3,6			-4	29	23	H,6,2		
-6	39	-41	8	32	-37	6	57	-55	H,2,14			4	76	-73	-2	34	-23	1	74	85
-5	34	45							-9	63	-55							3	31	-34
-4	74	87	H,C,6			H,1,6			-3	87	-71	H,3,4			H,4,15					
-3	121	142	-11	37	55	6	31	-20				-9	46	41	-6	30	17	H,6,3		
-1	75	76	6	72	78				H,2,15									4	35	29
0	89	-101				H,1,4			-2	31	23	H,3,3			H,5,13			0	54	-57
			H,C,8			7	34	-37	-4	35	33	-10	40	-33	-9	76	66	-3	51	-59
H,5,4			4	27	-36				-6	46	-35	-9	37	-31	-6	35	-34	-4	38	-46
2	34	-38				H,1,3			-7	80	-69	6	38	-30				-5	47	48
1	26	-15	H,C,10			7	61	-67	-9	50	35				H,5,12			-6	45	50
-1	139	140	-13	37	-53							H,3,1			-6	34	41			
-2	58	-63	2	32	30	H,1,2			H,2,16			-9	42	-32	-7	65	54	H,6,4		
-3	85	-93	4	37	-39	9	30	28	-6	28	22				-9	59	-58	-6	31	-37
-4	52	-66							-5	42	39	H,4,1						-5	71	-83
-6	37	45	H,C,12			H,1,1						7	36	28	H,5,11			-4	33	42
			-12	41	-48	-9	40	38	H,3,16			-9	38	33	-9	56	56	-3	100	118
H,5,3			0	36	40				-5	58	-52				-7	33	-32	-2	80	89
-5	78	82	1	72	83	H,1,0			-6	30	-22	H,4,2			-6	26	-12			
-4	105	120				9	32	63	-8	53	45	-8	81	-77	-5	35	31	H,6,5		
-3	88	103	H,C,14			-9	71	-69										-1	68	-76
-2	49	-50	-1	33	-50	-10	36	-17	H,3,15			H,4,3			H,5,10			-2	33	-38
1	85	-86							-9	31	-31	5	33	-30	-3	32	26	-3	41	-51
2	51	-56	H,C,16			H,2,2			-8	63	-44	-9	48	35				-4	40	-39
			-11	37	-46	-11	33	-28	-6	51	45				H,5,9			-5	30	38
H,5,2						-9	36	-36	-5	55	42	H,4,4			-10	31	-28	-6	73	84
1	62	-61	H,C,18									-9	40	-30	-9	49	-41	-7	30	36
0	31	27	-10	32	41	H,2,3			H,3,14			-8	45	-34	-7	43	-42			
-1	53	-67	-6	31	18	7	41	37	-3	88	74	5	72	71	-6	58	61	H,6,6		
-4	31	-33				-10	27	20	-4	33	-21				-5	74	78	-7	43	44
-5	64	-73	H,1,17						-5	44	-40	H,4,7			-4	76	88	-5	47	-50
			-11	32	24	H,2,4			-6	48	-38	3	40	-38	-1	58	-58	-4	32	-26
H,5,1			-10	34	-42	7	29	-18	-7	45	40	2	36	-31	0	37	-35	-3	38	40
-5	44	51							-11	34	-22	-9	104	-96	2	29	9	-2	34	44
-3	68	-74	H,1,16			H,2,5						-11	30	13						
-2	65	-68	-3	43	37	-10	30	15	H,3,13						H,5,8			H,6,7		
0	169	165	-8	28	-24				-10	50	-45	H,4,8			0	39	30	-2	71	-80
2	42	-42				H,2,6			-9	87	-79	-11	30	-22	-1	41	-40			
3	45	-50	H,1,15			-12	29	-15	-8	43	-44	-9	27	27	-6	86	84			
			-8	38	41				-4	40	-34				-8	86	-79			
H,5,0						H,2,7			-2	41	30	H,4,10								
2	80	55	H,1,14			5	30	-26				-9	55	-56	H,5,7					
1	149	159	0	34	34				H,3,12			-8	39	-36	-9	58	-57			
			-10	57	59				-1	45	-46	-1	26	-29	0	63	-64			

The *E*-map computed from the 232 terms, for which absolute signs could now be allotted, revealed the positions of all 12 non-hydrogen atoms of the molecule. The *R*-value derived from the structure postulated from this map was 0.42, and reduced to 0.34 after 5 cycles of Fourier refinement. The refinement was continued, using the full matrix least squares method. Again an overall scale factor and atomic coordinates were varied for all cycles. For the first 3 cycles, isotropic temperature factors were also varied. These were replaced by anisotropic parameters for a further 3 cycles, after which the *R*-value was 0.108. The weighting scheme and atomic scattering factors used were the same as those described for the *syn*-compound. A difference Fourier map was calculated at this stage, and it showed clearly all 11 hydrogen atoms of the molecule. As for the *syn*-compound, the contributions of the hydrogen atoms to the scattering were included in the remaining 2 cycles of least squares refinement, but their positional parameters and temperature factors of 4.0 Å^2 were not varied. The final *R*-value was 0.088.

Table 6. Fractional atomic coordinates for *anti*-ethyl benzohydroximate.

	x	y	z
C(1)	0.7139 (7)	0.9063 (10)	0.4560 (5)
C(2)	0.5763 (8)	0.9170 (12)	0.4306 (5)
C(3)	0.4784 (9)	1.0813 (15)	0.3680 (6)
C(4)	0.5264 (10)	1.2372 (13)	0.3344 (6)
C(5)	0.6641 (9)	1.2282 (11)	0.3624 (6)
C(6)	0.7618 (8)	1.0635 (10)	0.4234 (5)
C(7)	0.8142 (7)	0.7268 (09)	0.5213 (5)
N(8)	0.9007 (6)	0.6264 (08)	0.5132 (4)
O(9)	0.8949 (5)	0.6915 (06)	0.4257 (4)
O(10)	0.8009 (5)	0.6708 (07)	0.5964 (4)
C(11)	0.8742 (9)	0.4754 (11)	0.6575 (6)
C(12)	0.8239 (11)	0.4362 (14)	0.7199 (8)
H(2)	0.544	0.794	0.461
H(3)	0.366	1.084	0.345
H(4)	0.452	1.368	0.284
H(5)	0.701	1.355	0.335
H(6)	0.873	1.055	0.445
H(9)	0.969	0.606	0.417
H(111)	0.844	0.346	0.600
H(112)	0.995	0.490	0.709
H(121)	0.874	0.293	0.763
H(122)	0.710	0.450	0.680
H(123)	0.800	0.550	0.760

Table 7. Thermal parameters for *anti*-ethyl benzohydroximate. (The expression for the temperature factor is given in Table 4.)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	4.2 (4)	3.3 (3)	4.1 (3)	0.5 (3)	2.5 (3)	−0.2 (3)
C(2)	5.0 (4)	5.2 (4)	5.6 (4)	0.7 (4)	3.4 (4)	0.5 (4)
C(3)	6.3 (5)	7.1 (5)	7.1 (5)	2.8 (5)	4.2 (4)	1.0 (5)
C(4)	7.8 (5)	4.8 (5)	6.0 (4)	2.7 (4)	4.3 (4)	1.0 (4)
C(5)	7.2 (5)	3.5 (4)	5.7 (4)	0.6 (4)	3.4 (4)	−0.1 (3)
C(6)	5.5 (4)	3.1 (3)	5.1 (4)	0.1 (3)	3.1 (4)	−0.4 (3)
C(7)	4.9 (4)	2.8 (3)	4.4 (3)	0.0 (3)	2.8 (3)	0.0 (3)
N(8)	6.3 (3)	2.4 (3)	6.1 (3)	0.6 (2)	4.5 (3)	0.1 (2)
O(9)	7.1 (3)	3.4 (2)	7.0 (2)	1.1 (2)	5.3 (3)	0.8 (2)
O(10)	7.8 (3)	3.4 (2)	6.5 (2)	2.0 (2)	4.9 (3)	1.4 (2)
C(11)	7.7 (5)	4.0 (4)	6.4 (4)	1.7 (3)	4.3 (4)	1.7 (4)
C(12)	12.6 (7)	6.6 (5)	11.8 (7)	4.4 (5)	10.3 (7)	4.7 (5)

Observed and calculated structure factors are listed in Table 5. Table 6 lists the fractional coordinates of the heavier atoms and the approximate fractional coordinates of the hydrogen atoms. Table 7 lists the thermal parameters of the non-hydrogen atoms.

Most of the calculations were performed on the IBM 7094 computer at NEUCC, Lundtofte, Denmark, using mainly the integrated program system *X-RAY-63*.[11] In addition were used a least squares programme for refinement of the cell parameters, as described by Liminga,[12] and the series of programmes

for executing the symbolic addition procedure for centrosymmetric structures, written by Dewar.[13] Programmes to produce an input tape to, and to process the output tape from the diffractometer have been written by Sørensen.[14]

DISCUSSION

The X-ray investigation showed that the configurations proposed by Exner *et al.*[2] were correct, the compound with the lower melting point (53°) being the *syn*-isomer (*i.e.* *Z*-ethyl benzohydroximate), and the compound with the higher melting point (67°) being the *anti*-isomer (*i.e.* *E*-ethyl benzohydroximate).

The geometry of the molecules is illustrated in Fig. 1. The benzene ring of both molecules is planar within the experimental error, and the coplanarity includes the carbon atom C(7). In addition, the atoms C(1), C(7), N(8), O(9), and O(10) of the side chains are nearly coplanar, as expected. Deviations from the coplanarity, expressed as distances of the atoms from the planes

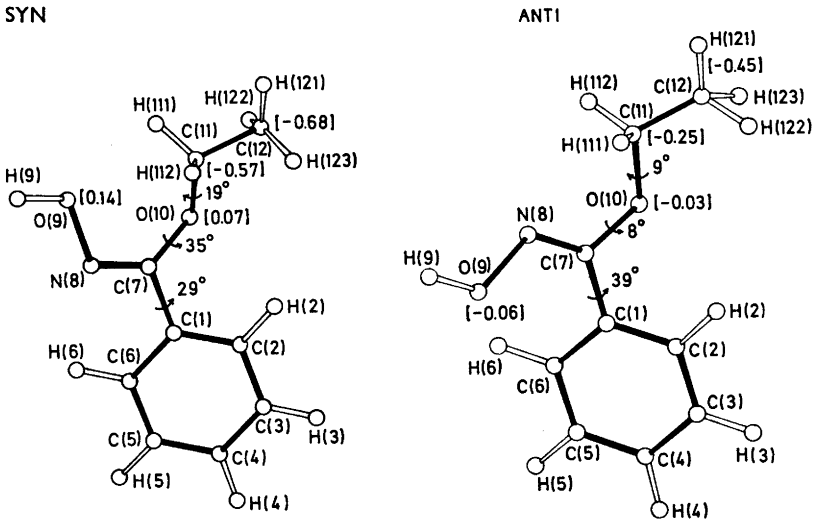

Fig. 1. Description of the conformations of *syn*- and *anti*-ethyl benzohydroximate. The numbers in the angular parentheses are the distances (Å) from the planes defined by C(1), C(7), and N(8). In addition, the dihedral angles of the C(1)–C(7), C(7)–O(10), and O(10)–C(11) bonds are given.

defined by C(1), C(7), and N(8), are given in Fig. 1. The angles between the two planes are 29° and 39° for the *syn*- and *anti*-form, respectively. This twist is in both cases larger than needed, to relieve the steric strain between the side chain and the hydrogen atom in the *ortho* position of the benzene ring, and is probably determined by the packing of the molecules rather than by steric interference effects within the molecule.

The carbon atoms of the ethyl groups are not coplanar with the aforenamed atoms of the side chains. This is indicated in Fig. 1, where the dihedral angles of the C(7) – O(10) bonds are given. The larger angle of the *syn*-compound is probably due to steric strain between the methylene group and the hydroxyl group. The *intra*-molecular distance between C(11) and O(9) is 2.74 Å. In both compounds, the $O-C_{alkyl}$ bond is situated approximately *s-cis* to the C=N bond. This conformation is in accordance with the stable conformation of carboxylic esters.[15] The ethyl group of both isomers has the preferred staggered conformation.

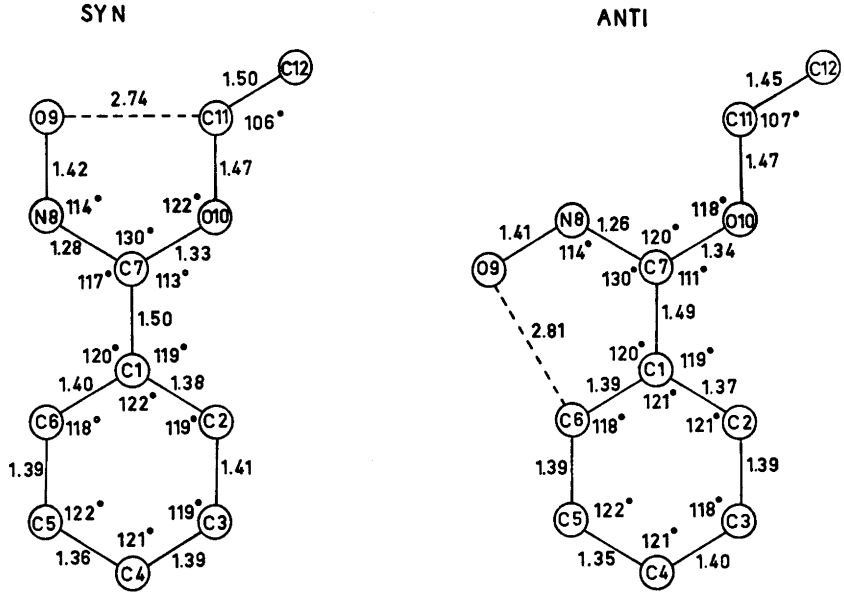

Fig. 2. Intra-molecular distances (Å) and angles (°) for *syn*- and *anti*-ethyl benzohydroximate. Dashed lines indicate short, non-bonded distances. The standard deviations on the bond lengths are 0.007 – 0.021 Å, and on the angles 0.5 – 1.0°.

Bond lengths and valency angles of both molecules are shown in Fig. 2. There is good agreement between corresponding bonds and angles of the two isomers, all differences being less than three times the estimated standard deviations. The only exception is the valency angles of the trigonal hybridized carbon atom C(7). In the *syn*-form, the angle O(10) – C(7) – N(8) is expanded to 130°, whereas in the *anti*-form, the angle C(1) – C(7) – N(8) is 130°. In both cases, the expansions from the ideal 120° can be ascribed to van der Waals strain between the oxygen atom of the hydroxyl group and another part of the molecule, *i.e.* in the *syn*-form, the methylene group, C(11), and in the *anti*-form, the *ortho* hydrogen atom, H(6).

In addition, the dimensions of the oxime moiety of the molecules, –C–C=N–OH, agree remarkably well with those found in the crystal structures of some benzenoid oximes, *e.g. syn*- and *anti-p*-chlorobenzaldoxime.[16]

The lengths of the bonds C–C, C=N, and N–O, and the angle C–N–O are identical within the experimental error. The opening of the C–C=N angle of the *anti*-compound mentioned above was also observed in the *anti*-forms of the oximes.[16] This seems to be the normal way of relieving van der Waals

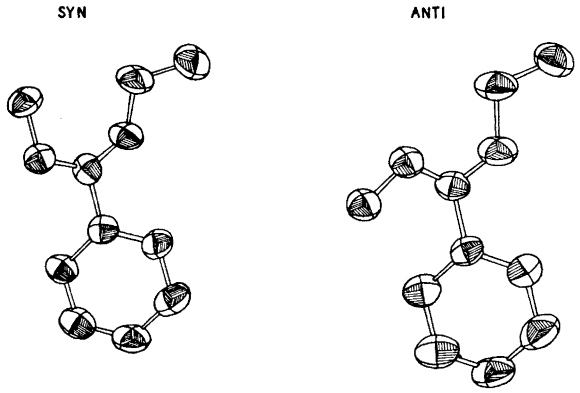

Fig. 3. Vibrational ellipsoids at the 50 % level for *syn*- and *anti*-ethyl benzohydroxymate, prepared from the computer program of Johnson.[19]

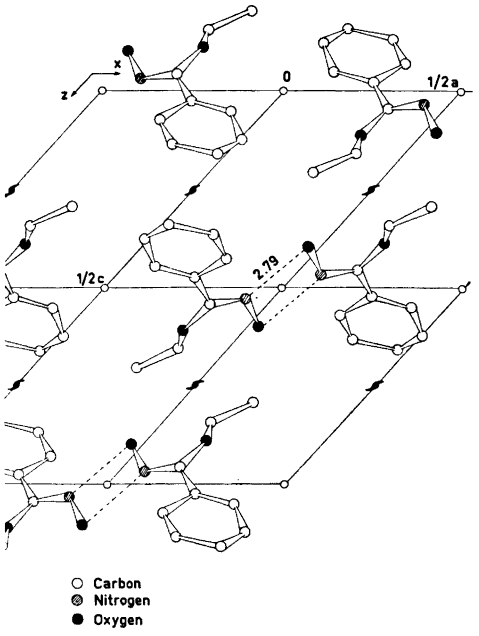

Fig. 4. Projection of the structure of *syn*-ethyl benzohydroxymate along the *b*-axis.

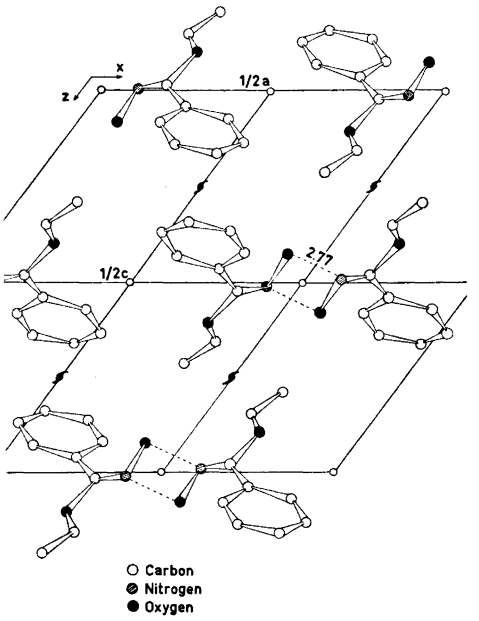

Fig. 5. Projection of the structure of *anti*-ethyl benzohydroxymate along the *b*-axis.

strain, whereas the torsional angles about the $C_{arom.}-C_{oxime}$ bond show considerable variations, probably determined by the packing conditions in the appropriate crystal.

The dimensions of the ethoxy groups do not differ significantly from the values given for carboxylic esters [15] or ethyl carbamate,[17] although the ester groups of these molecules are planar.

The relatively high standard deviations on bond lengths and angles (Fig. 2) are probably due to the large atomic vibration of both molecules (*cf.* Table 6). The directions of the vibrational ellipsoids are illustrated in Fig. 3. As expected, the terminal methyl C-atoms have the largest thermal motion.

The molecular arrangement in the crystals of both compounds is dominated by OH···N hydrogen bonds, which in both cases couples the molecules to dimers around centers of symmetry (*cf.* Figs. 4–5). A similar system of hydrogen bonding is found to be quite normal for aromatic *syn*-oximes, whereas *anti*-oximes normally are linked to infinite chains by OH···N hydrogen bonds.[18] No other short *inter*-molecular distances were found in the crystal structures of the compounds.

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